

RESERVE COPY
PATENT SPECIFICATION



Application Date : Nov. 10, 1933. No. 31339/33.

428,462

Complete Specification Left: Oct. 15, 1934.

Complete Specification Accepted: May 10, 1935.

PROVISIONAL SPECIFICATION.

Improved Manufacture of Derivatives of Glycerol

I, ERIC LEIGHTON HOLMES, of the Chemical Research Laboratory, Teddington, Middlesex, a British Subject, do hereby declare the nature of this invention to be as follows:—

The object of my invention is to prepare derivatives of glycerol and for this purpose I used dehydrogenating catalysts and preferably those containing copper.

10 In accordance with my invention I subject glycerol to the action of the heated dehydrogenating catalyst and collect the product which might either be used as such, or fractionated to give the products
15 described later.

The following examples describing the use of dehydrogenating catalysts containing copper illustrate but do not limit my invention. The copper chromite catalyst
20 referred to in example 1 is conveniently that used by Adkins and Connor, J.A.C.S., 1931, 53, 1091—5.

EXAMPLE 1.

125 g. of glycerol at room temperature
25 were dropped on to 20 cc. of copper chromite catalyst in a glass tube of 2.5 cm. diameter at 240—260°C. and at a rate of 1 drop per second.

Yield = 115 g. of a light brown limpid
30 liquid which when distilled under atmospheric pressure gave the following fraction, among others:—

B.p. 140—150°C. (41 g.) phenylhydrazones m.p. 140—142° together with a residue containing unchanged glycerol 35 and tarry matter. This fraction, the main product, redistilled as a pale yellow liquid B.p. 144—146°.

EXAMPLE 2.

100 g. of glycerol were dropped on to 40 60 cc. of copper oxide wire, which had been reduced in a stream of hydrogen, at a rate of 1 drop per 3 seconds and at a temperature of 240—260°C. Distillation of the rather dark product yielded sub- 45 stantially the same products as in Example 1, except that the residue amounted to over 50 per cent., indicating a lower rehydrogenating efficiency.

The main product of the reaction, B.p. 50 144—146°, has good solvent powers for cellulose derivatives.

Absolute proof of its constitution has not yet been obtained, though it is reduced catalytically to an alcohol boil- 55 ing at 182—4°.

The temperature of working may vary from about 200°C. to about 300°C. With the catalysts referred to in Examples 1 and 2 the optimum temperature seems to 60 be about 260° C.

Dated the 10th day of November, 1933.

ERIC LEIGHTON HOLMES.

COMPLETE SPECIFICATION

Improved Manufacture of Derivatives of Glycerol

I, ERIC LEIGHTON HOLMES, of the Chemical Research Laboratory, Teddington, Middlesex, a British Subject, do
65 hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

70 It is known that when glycerol is heated at 450°C in a tube filled with pumice it is decomposed into several products among which is acetol.

It is also known that when vapours of
75 glycerol are passed at 330°C over copper prepared by the reduction of copper

carbonate at a low temperature, the initial effect is the dehydrogenation of the glycerol to glyceric aldehyde which
80 decomposes into ethyl alcohol and carbon dioxide; there is also some dehydration of the glycerol to acroleine; these two products undergo further decomposition, condensation or hydrogenation but acetol
85 has not been named as occurring among the products.

This invention consists in the manufacture of a liquid consisting chiefly of acetol and of value as a solvent for
90 cellulose derivatives, by subjecting liquid glycerol to the action of a dehydrogenat-

[Price 1/-]

Price 4s 6d

ing catalyst which may or may not have also a dehydration effect and is preferably one containing copper, heated to a temperature between 200 and 300°C, and collecting the liquid produced by condensing the vapours evolved. By fractional distillation the liquid yields a fraction which boils at 140—150°C and when redistilled gives substantially pure acetol, which differs from the acetol hitherto known in that it does not readily polymerise on standing.

The following Examples illustrate the invention:—

EXAMPLE 1.

125 grams of glycerol at room temperature are dropped on to 20 cc. of copper chromite catalyst (which may be made as described by Adkins & Conner in Journal of the American Chemical Society 1931, 53, pages 1091—5), contained in a glass tube of 2.5 cm. diameter heated to 240—260°C., at a rate of 1 drop per second. The vapours leaving the tube are passed into a condenser whereby 115 grams of a light brown limpid liquid are obtained. When this is fractionally distilled under atmospheric pressure, the chief fraction (41 grams) boils at 140—150°C. By redistilling this fraction substantially pure acetol, boiling at 144—146°C., is obtained.

EXAMPLE 2.

100 grams of glycerol are dropped, at the rate of 1 drop per 3 seconds, on to 60 cc. of copper oxide wire which has been reduced by heating it in a stream of hydrogen and is at a temperature of 240—260°C; the product is worked up as

described in Example 1, but the yield is considerably less than in that Example.

Having now particularly described and ascertained the nature of my said invention and in what manner the same is to be performed, I declare that what I claim is:—

1. A manufacture of a solvent which consists in subjecting liquid glycerol to the action of a dehydrogenating catalyst heated to a temperature between 200°C and 300°C and condensing to a liquid the vapours thus produced.

2. A manufacture of acetol wherein liquid glycerol is subjected to the action of a dehydrogenating catalyst heated to a temperature between 200°C and 300°C, the vapours thus produced are condensed to a liquid and this liquid is fractionally distilled to obtain a fraction that boils at 140—150°C, which is redistilled.

3. A manufacture as claimed in Claim 1 or 2, wherein the catalyst is one containing copper and is heated to 240—260°C.

4. A process of subjecting glycerol to the action of a heated catalyst substantially as described in either of the Examples herein.

5. Acetol which does not readily polymerise on standing, when produced by the process of manufacture particularly described and ascertained herein.

Dated this 15th day of October, 1934.
ABEL & IMRAY,
30, Southampton Buildings,
London, W.C.2,
Agents for the Applicant.